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28-DAY COMPRESSIVE STRENGTH MONITOR CEMENT SAMPLE CM-2

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by

G.G. Carette*, K.E. Painter** and W.S. Bowman***

INTRODUCTION

Following the depletion of the first National Strength Monitor Cement Sample, CM-1, a second National Monitor Cement Sample on Strength has been prepared under the auspices of the Canadian Standards Association (CSA) Committee on Hydraulic Cements. It was packaged by the Canadian Certified Reference Materials Project (CCRMP) who will also look after storage and sales. A Canadian interlaboratory test program was carried out by the Mineral Sciences Laboratories (MSL) to determine the 28-day cube compressive strength of mortars incorporating this material using the CSA procedure described in CAN-3-A5-M88. No other property was evaluated. CCRMP performed statistical analysis of the interlaboratory data and the results are presented herein.

ORIGIN AND PREPARATION OF CM-2

Sample CM-2 is a Type 20 cement obtained from the production of a Canadian cement plant. About 1.5 metric tonne of the material taken from a given production lot was blended at the Conchem production facilities in Milton, Ontario. The blender used was a heavy duty drive ribbon-type blender of 1.6 m^3 total capacity, and capable of handling up to 2 metric tonnes of cement. The material was blended in one lot for about an hour which, based on similar experiences, was sufficient to insure adequate mixing and homogeneity of the material. The blended material was shipped to CANMET for bagging into 2.5 kg units and packing into plastic containers for distribution by CCRMP. Twenty-two of these blended units were then used for strength characterization of the material through an interlaboratory CANMET-sponsored test program.

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INTERLABORATORY TEST PROGRAM

Twenty-two Canadian cement testing laboratories participated in the cooperative testing program used to characterize cement sample, CM-2, by testing its 28-day cube compressive strength in mortars. These laboratories are listed herewith alphabetically. Each was assigned a code number so that analytical results could be recorded while preserving the anonymity of the laboratory. The code number bears no relationship to the alphabetical order of the laboratories.

Participating Laboratories

Ciment Quebec Inc., St. Basile, Quebec
Inland Cement Limited, Edmonton, Alberta
Inland Cement Limited, Regina, Saskatchewan
Inland Cement Limited, Winnipeg, Manitoba
Lafarge Canada Inc., Bath, Ontario
Lafarge Canada Inc., Brookfield, Nova Scotia
Lafarge Canada Inc., Exshaw, Alberta
Lafarge Canada Inc., Kamloops, British Columbia
Lafarge Canada Inc., Montreal, Quebec
Lafarge Canada Inc., Richmond, British Columbia
Lafarge Canada Inc., St. Constant, Quebec
Lafarge Canada Inc., Woodstock, Ontario
Lake Ontario Cement, Picton, Ontario
Ministry of Transportation of Ontario, Downsview, Ontario
North Star Cement Limited, Corner Brook, Newfoundland
Ontario Hydro, Toronto, Ontario
St. Lawrence Cement Company, Beauport, Quebec
St. Lawrence Cement Company, Joliette, Quebec
St. Lawrence Cement Company, Mississauga, Ontario
St. Marys Cement Co., Bowmanville, Ontario
St. Marys Cement Co., St. Marys, Ontario
Tilbury Cement Limited, Delta, British Columbia

Two samples of the homogenized cement material were sent to each of the participating laboratories who were asked to determine the compressive strength of mortar cubes as per CAN/CSA-AS-M88. For each of the two samples provided, they were asked to perform two sets of strength determination on two duplicate mortar batches prepared within a period not exceeding 72 h. Each set of strength determinations was to include three cubes to be broken in compression at the age of 28 days. The two samples were to be tested within a period not exceeding two weeks and, as far as possible, the same and most experienced operator was to perform all tests. The tests were to be carried out using a constant water-to-cement ratio of 0.485.

All twenty-two participating laboratories completed the four sets of strength determinations as requested. The data as received by MPL was compiled and submitted to CCRMP for evaluation.

STATISTICAL EVALUATION OF THE TEST DATA

The test data, their means and standard deviations are reported in Table 1. They are also plotted in Figure 1. The 'x' in Figure 1 represent measurements on the first test sample while the '+' are for measurements on the second sample. It is readily apparent from the figure that several laboratories got significantly different results for the two samples. A significant difference between samples was confirmed by a statistical test using a two-way ANOVA[1]. Because of the blending which had been done, it was felt that the observed sample differences were not likely due to inhomogeneity but, rather, to slight variations in the test procedure. It was also felt that the differences were negligible compared to the between-laboratory variation. There was no evidence of outlying results, either in terms of high variance or in being remote from the consensus. Thus, all data were used for computations. A one-way analysis of variance technique (ANOVA) was used to estimate the consensus value and its variance. This technique assumes that the test data are a sampling of a universal set of results. They are assumed to fit the following model:[1]

$$x_{ij} = \mu + y_i + e_{ij}$$

where x_{ij} = the j^{th} result in set i,
μ = the true consensus value,
y_i = the discrepancy between the mean of the results in the set i ($\bar{x}_{i\cdot}$) and μ
and e_{ij} = the discrepancy between x_{ij} and $\bar{x}_{i\cdot}$.

It is assumed that both y_i and e_{ij} are normally distributed with means of zero and variances of σ^2 and ω^2, respectively. The significance of ω^2 is detected by comparing the ratio of between-sets mean squares to within-set mean squares, with the F statistic at the 95% confidence level and with the appropriate degrees of freedom. The consensus value of the assumed model is estimated by the overall mean:

$$\bar{x}_{\cdot\cdot} = \sum_{i}^{k} \sum_{j}^{kn_i} x_{ij} \Big/ \sum_{i}^{k} n_i$$

where n_i = the number of results in set i,
and k = the number of sets.

The value of σ^2 is estimated by s_1^2, which is given by:

$$s_1^2 = \sum_i^k \sum_j^{n_i} (x_{ij} - \bar{x}_{i\cdot})^2 / (\sum_i^k n_i - k).$$

The value of ω^2 is estimated by:

$$\omega^2 = (k-1) \cdot (s_2^2 - s_1^2) / (\sum_i^k n_i - \sum_i^k n_i^2 / \sum_i^k n_i)$$

where

$$s_2^2 = \sum_i^k n_i (\bar{x}_{i\cdot} - \bar{x}_{\cdot\cdot})^2 / (k-1).$$

The variance of the overall mean is given by:

$$V[\bar{x}_{\cdot\cdot}] = (\sum_i^k n_i^2 / (\sum_i^k n_i)^2) \omega^2 + \sigma^2 / \sum_i^k n_i$$

and the 95% confidence limits for $\bar{x}_{\cdot\cdot}$ are:

$$\bar{x}_{\cdot\cdot} \pm t_{0.975,(k-1)} \{V[\bar{x}_{\cdot\cdot}]\}^{1/2}.$$

It should be noted that 95% confidence limits denote that if the certification program were performed 100 times, the overall mean in 95 instances would fall within the prescribed limits.

Criterion for Certification

The ratio of the between-laboratories to the within-laboratory standard deviation, σ_B/σ_A, where

$$\sigma_B = \{ \sum_i^k (\bar{x}_{i\cdot} - \bar{x}_{\cdot\cdot})^2 / (k-1) \}^{1/2}$$

is a measure of the quality of the certification data used by CCRMP (2). σ_A is the unweighted average of the laboratory standard deviations in Table 1. σ_A is numerically equal to S_1 provided that each laboratory reports the same number of replicates, as is the case here. The acceptable upper limit of σ_B/σ_A is 3. The criterion for certification is RP, the percentage of sets of results that must be removed to yield a value of σ_B/σ_A equal to less than the acceptable upper limit. The value of RP may not exceed 15% for an element to be considered certifiable. When this is true, the overall mean is assigned as the recommended value.

The recommended value of 28-day compressive strength and other statistical parameters are reported in Table 2. Since the criteria for certification for a CCRMP reference material have been met, CM-2 will be made available for purchase as a reference monitor cement.

REFERENCES

1. Brownlee, K.A. Statistical Theory and Methodology in Science and Engineering; John Wiley and Sons, Inc.; New York; 1960.

2. Steger, H.F. "A re-assessment of the criteria for certifiability in CCRMP"; Geo-standards Newsletter 6:1:17-23; 1982.

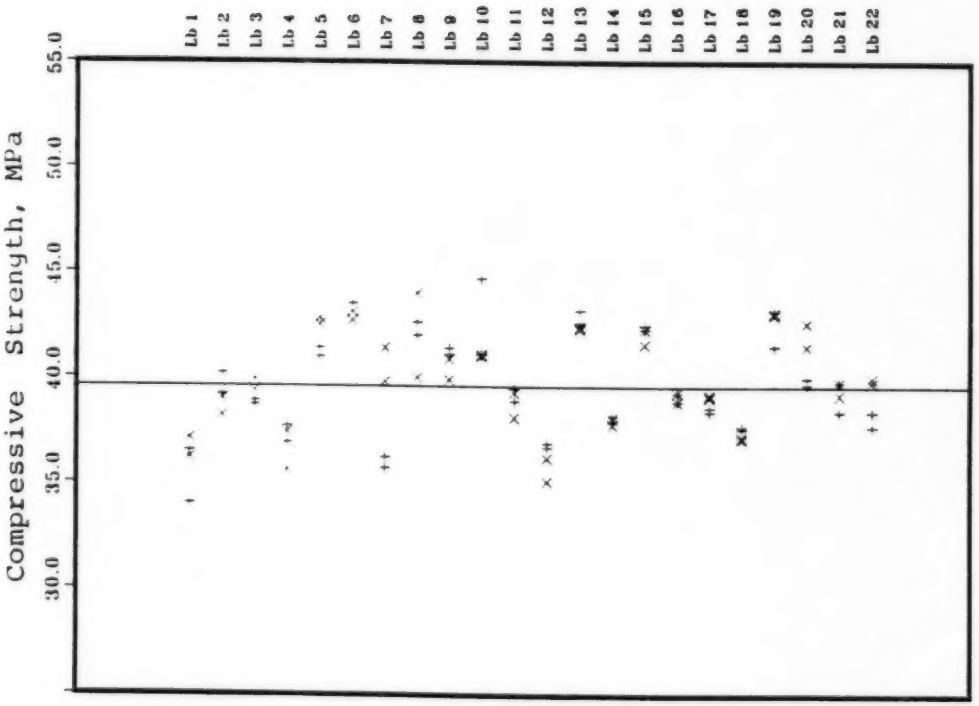
Table 1. CSA/CCRMP Monitor Cement CM-2 ----- 28-Day Compressive Strength, MPa

Laboratory	Sample 1		Sample 2		Mean	S.D.
Lab- 1	37.1	36.2	36.5	34.0	35.95	1.35
Lab- 2	39.0	38.2	40.2	39.2	39.15	0.82
Lab- 3	39.4	39.9	38.7	38.9	39.23	0.54
Lab- 4	35.6	37.4	36.9	37.7	36.90	0.93
Lab- 5	42.5	42.8	41.4	41.0	41.93	0.86
Lab- 6	43.1	42.7	43.5	43.5	43.20	0.38
Lab- 7	41.4	39.8	35.7	36.2	38.28	2.77
Lab- 8	40.0	44.0	42.0	42.6	42.15	1.66
Lab- 9	40.9	39.9	41.4	41.1	40.82	0.65
Lab-10	41.1	41.0	44.7	41.1	41.98	1.82
Lab-11	39.3	38.1	38.9	39.5	38.95	0.62
Lab-12	35.1	36.2	36.9	36.7	36.23	0.81
Lab-13	42.4	42.3	43.2	42.6	42.63	0.40
Lab-14	38.1	37.8	38.0	38.2	38.03	0.17
Lab-15	41.6	42.3	42.3	42.5	42.18	0.39
Lab-16	39.3	38.9	38.9	39.3	39.10	0.23
Lab-17	39.1	39.2	38.6	38.4	38.83	0.39
Lab-18	37.1	37.2	37.7	37.6	37.40	0.29
Lab-19	43.1	43.0	43.2	41.5	42.70	0.80
Lab-20	41.5	42.6	39.7	40.0	40.95	1.35
Lab-21	39.2	39.8	39.8	38.4	39.30	0.66
Lab-22	40.0	39.8	37.7	38.4	38.98	1.11

(Each value is the average of 3 mortar cubes)

Table 2. Recommended Value and Associated Statistical Parameters for CSA/CCRMP Monitor Cement CM-2 --- 28-day Compressive Strength

No. of laboratories	22	
No. of sets of results	22	
Recommended Value	39.8	MPa
95% confidence limits		
low	38.8	MPa
high	40.7	MPa
σ	1.1	MPa
ω	2.1	MPa
Certification criterion	2.6	
Rejection Percentage	0.0	



CSA/CCRMP Monitor Cement CM-2 --- 28-Day Compressive Strength, MPa
Sample: CM-2

Figure 1.